

§91.316

40 CFR Ch. I (7–1–07 Edition)

§91.316 Hydrocarbon analyzer calibration.

(a) Calibrate the FID and HFID hydrocarbon analyzer as described in this section. Operate the HFID to a set point $\pm 5.5^{\circ}\text{C}$ between 185 and 197 $^{\circ}\text{C}$.

(b) Initial and periodic optimization of detector response. Prior to introduction into service and at least annually thereafter, adjust the FID and HFID hydrocarbon analyzer for optimum hydrocarbon response as specified by this paragraph. Alternative methods yielding equivalent results may be used, if approved in advance by the Administrator.

(1) Follow good engineering practices for initial instrument start-up and basic operating adjustment using the appropriate fuel (see §91.312) and purified synthetic air or zero-grade nitrogen.

(2) One of the following procedures is required for FID or HFID optimization:

(i) The procedure outlined in Society of Automotive Engineers (SAE) paper No. 770141, "Optimization of Flame Ionization Detector for Determination of Hydrocarbons in Diluted Automobile Exhaust"; author, Glenn D. Reschke. This procedure has been incorporated by reference. See §91.6.

(ii) The HFID optimization procedures outlined in 40 CFR part 1065, subpart D.

(iii) Alternative procedures may be used if approved in advance by the Administrator.

(3) After the optimum flow rates have been determined, they are recorded for future reference.

(c) Initial and periodic calibration. Prior to introduction into service and monthly thereafter, or within one month prior to the certification test, calibrate the FID or HFID hydrocarbon analyzer on all normally used instrument ranges, using the steps in this paragraph. Use the same flow rate and pressures as when analyzing samples. Introduce calibration gases directly at the analyzer. An optional method for dilute sampling described in 40 CFR part 1065, subpart F, may be used.

(1) Adjust analyzer to optimize performance.

(2) Zero the hydrocarbon analyzer with purified synthetic air or zero-grade nitrogen.

(3) Calibrate on each used operating range with calibration gases having nominal concentrations between 10 and 90 percent of that range. A minimum of six evenly spaced points covering at least 80 percent of the 10 to 90 percent range (64 percent) is required (see following table).

Example calibration points (percent)	Acceptable for calibration?
20, 30, 40, 50, 60, 70	No, range covered is 50? percent, not 64 percent.
20, 30, 40, 50, 60, 70, 80, 90 ..	Yes.
10, 25, 40, 55, 70, 85	Yes
10, 30, 50, 70, 90	No, though equally spaced and entire range covered, a minimum of six points is needed

(4) For each range calibrated, if the deviation from a least-squares best-fit straight line is two percent or less of the value at each data point, calculate concentration values by use of a single calibration factor for that range. If the deviation exceeds two percent at any point, use the best-fit non-linear equation which represents the data to within two percent of each test point to determine concentration.

(d) Oxygen interference optimization. Choose a range where the oxygen interference check gases will fall in the upper 50 percent. Conduct the test, as outlined in this paragraph, with the oven temperature set as required by the instrument manufacturer. Oxygen interference check gas specifications are found in §91.312(d).

(1) Zero the analyzer.

(2) Span the analyzer with the 21 percent oxygen blend.

(3) Recheck zero response. If it has changed more than 0.5 percent of full scale repeat paragraphs (d)(1) and (d)(2) of this section to correct the problem.

(4) Introduce the 5 percent and 10 percent oxygen interference check gases.

(5) Recheck the zero response. If it has changed more than ± 1 percent of full scale, repeat the test.

(6) Calculate the percent of oxygen interference (designated as percent O_2 I) for each mixture in paragraph (d)(4) of this section according to the following equation:

percent O_2 I = $(B - \text{Analyzer response (ppm C)})/B \times 100$

$$\text{analyzer response} = \left(\frac{A}{\% \text{ of fullscale analyzer response due to A}} \right) \times (\% \text{ of fullscale analyzer response due to B})$$

Where:

A=hydrocarbon concentration (ppmC) of the span gas used in paragraph (d)(2) of this section.

B=hydrocarbon concentration (ppmC) of the oxygen interference check gases used in paragraph (d)(4) of this section.

(7) The percent of oxygen interference (designated as percent O₂ I) must be less than ±three percent for all required oxygen interference check gases prior to testing.

(8) If the oxygen interference is greater than the specifications, incrementally adjust the air flow above and below the manufacturer's specifications, repeating paragraphs (d)(1) through (d)(7) of this section for each flow.

(9) If the oxygen interference is greater than the specification after adjusting the air flow, vary the fuel flow and thereafter the sample flow, repeating paragraphs (d)(1) through (d)(7) of this section for each new setting.

(10) If the oxygen interference is still greater than the specifications, repair or replace the analyzer, FID fuel, or burner air prior to testing. Repeat this section with the repaired or replaced equipment or gases.

[61 FR 52102, Oct. 4, 1996, as amended at 70 FR 40451, July 13, 2005]

§91.317 Carbon monoxide analyzer calibration.

(a) Calibrate the NDIR carbon monoxide analyzer described in this section.

(b) Initial and periodic interference check. Prior to its introduction into service and annually thereafter, check the NDIR carbon monoxide analyzer for response to water vapor and CO₂.

(1) Follow good engineering practices for instrument start-up and operation. Adjust the analyzer to optimize performance on the most sensitive range to be used.

(2) Zero the carbon monoxide analyzer with either purified synthetic air or zero-grade nitrogen.

(3) Bubble a mixture of three percent CO₂ in N₂ through water at room temperature and record analyzer response.

(4) An analyzer response of more than one percent of full scale for ranges above 300 ppm full scale or more than three ppm on ranges below 300 ppm full scale requires corrective action. (Use of conditioning columns is one form of corrective action which may be taken.)

(c) Initial and periodic calibration. Calibrate the NDIR carbon monoxide analyzer prior to its introduction into service and monthly thereafter.

(1) Adjust the analyzer to optimize performance.

(2) Zero the carbon monoxide analyzer with either purified synthetic air or zero-grade nitrogen.

(3) Calibrate on each used operating range with carbon monoxide-in-N₂ calibration gases having nominal concentrations between 10 and 90 percent of that range. A minimum of six evenly spaced points covering at least 80 percent of the 10 to 90 range (64 percent) is required (see following table).

Example calibration points (percent)	Acceptable for calibration?
20, 30, 40, 50, 60, 70	No, range covered is 50 percent, not 64 percent.
20, 30, 40, 50, 60, 70, 80, 90 ..	Yes.
10, 25, 40, 55, 70, 85	Yes.
10, 30, 50, 70, 90	No, though equally spaced and entire range covered, a minimum of six points is needed.

(4) Additional calibration points may be generated. For each range calibrated, if the deviation from a least-squares best-fit straight line is two percent or less of the value at each data point, concentration values may be calculated by use of a single calibration factor for that range. If the deviation exceeds two percent at any point, use the best-fit non-linear equation which represents the data to within